Mechanical characterization of multicrystalline silicon wafers

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1 Introduction

- Photovoltaic (PV) industry has a growth rate of 30%, a situation that many other industries can only dream about. Today, the 90% of the photovoltaic market share is covered by silicon solar where **multicrystalline silicon (mc-Si) represents the majority of all available commercial solar cells**. Furthermore, it will take at least a decade before any other PV technology based on other materials will become competitive.
- The dramatic growth in the PV industry has caused a **lack of solar-grade silicon** *-silicon with the required chemical purity for PV applications-*, resulting in increased prices for such material (silicon feedstock, crystallization and multi-wire sawing processes represent the 50% of the total solar module cost).
- Developing best quality and less expensive mc-Si cells can be achieved by reducing the wafers thickness, by recycling silicon from silicon containing waste (produced in large quantities during PV production), and by directly incorporating so far discarded silicon feedstock.
- Both strategies require exhaustive researches about the **effect of the higher concentration of impurities and their interaction with crystallographic defects** to reduce wafer thickness, in order to arrive at sufficiently high solar cell efficiencies and yields in solar cell and module production.

2 Materials and Methods

MATERIALS

- Silicon wafers tested were provided from a solar grade multi-crystalline silicon (mc-Si) block performed by the Vertical Gradient Freeze crystallization variant method.
- Mc-Si ingots were cut from the block and sliced with the multi-wire slurry saw into 250 µm and 2 mm thick wafers.
- Wafers from the middle and the top part of the ingots were collected.

METHODS

- Microstructure and fracture surface analysis: optical microscope and scanning electron microscope.
- Mechanical characterization: Young's modulus, flexure strength and fracture toughness with Three-point and Ring on ring bending tests.
- *Nanocharacterization*: nanoindentation on polished and SECCO etched probes.

3 Results

- **Three point flexural test**
- **Ring on Ring test**

— **Characteristic Strength σ_r (MPa)**

- **Dependence on specimen's thickness:** ↑σr → ↓ thickness
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- **Dependence on the part of the block:**
  - ROR: ↓σr → ↑ Position (due to the concentration of impurities)
  - TPB: ↓σr → ↓ Position

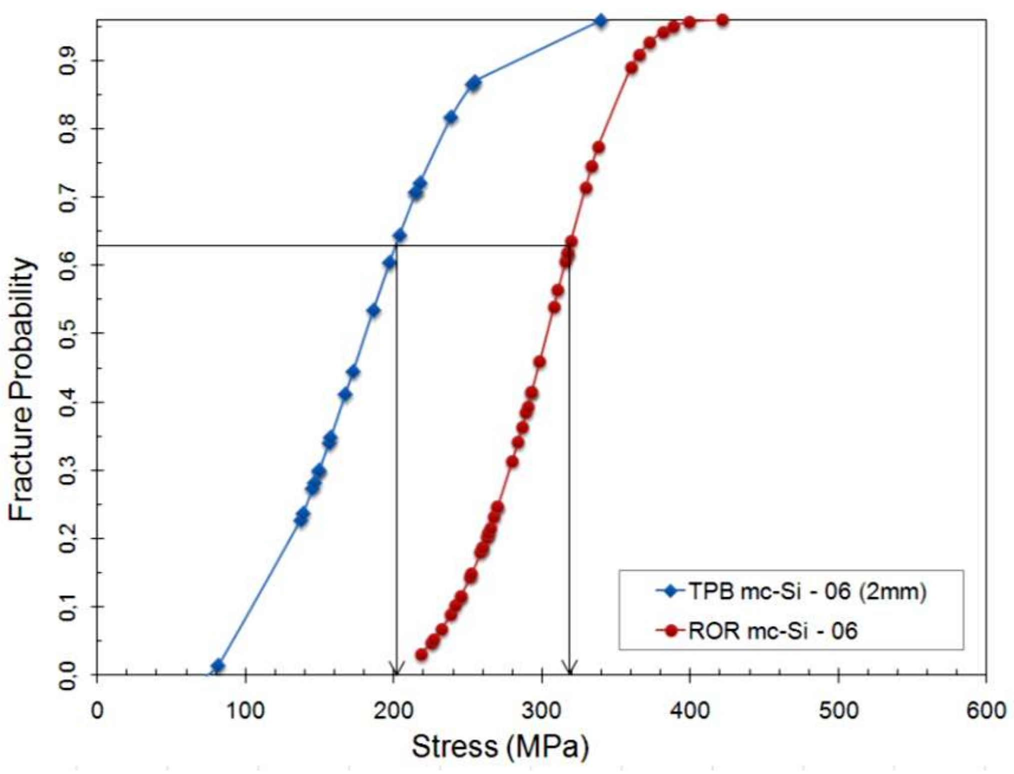

Figure 2. Weibull graph plotting fracture probability against applied stress with both techniques for mc-Si wafers collected from the top of the ingot

Table1. Statistical breakage rate of mc-Si wafers after TPB and ROR tests

Test / Wafer Position	Thickness (µm)	Characteristic strength, θ (MPa)	Weibull flexural modulus, F
ROR mc-Si – 03	250	380	3,930
3PBT mc-Si – 03	250	108	3,316
3PBT mc-Si – 03	2000	183	3,924
ROR mc-Si – 06	250	314	7,176
3PBT mc-Si – 06	2000	195	3,306

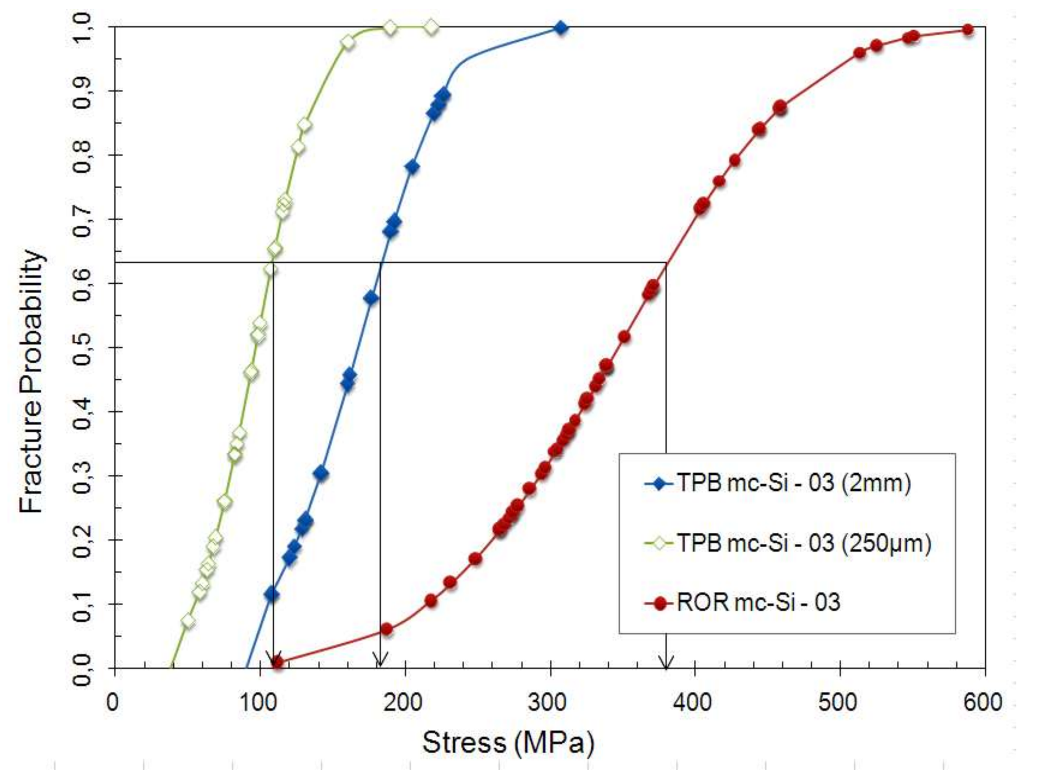

Figure 1. Weibull graph plotting fracture probability against applied stress with both techniques for mc-Si wafers collected from the middle of the ingot

Conventional single-edge-notched beam method
(180 µm and 35% depth straight notch cut with diamond wire)

Fracture Toughness — ***Middle*** : 1,36 ± 0,05 MPa.m$^{1/2}$; ***Top*** : 1,46 ± 0,07 MPa.m$^{1/2}$

Top wafers precipitates slightly act as a toughening agent

Nanoindentation (MaxLoad: 500 mN)
- Silicon *nanohardness* ~ 11,0 ± 0,5 GPa
- Silicon Carbide areas *nanohardness* ~ 13 ± 1 GPa
- NanoYoung Modulus ~ 185 ± 5 MPa

Flexural vibration resonance method
- Young Modulus ~ 175 ± 8 MPa

Not dependence on the part of the block analyzed or the impurities concentration

4 Fractographical analysis

Fracture initiation ↓ **Critical Surface defects** — Precipitates resulted from processing (inclusions and inhomogeneous distribution of the phases in the material) — Aluminum and small particles of silicon carbide (SiC)

I. An outer layer which shows a characteristic brittle fracture, hence the crack was initiated.
II. An inner layer, where the fracture progress quickly and cause and the plastic deformation of a small surface, prior to fracture and
III. A central zone subjected to a violent fracture.

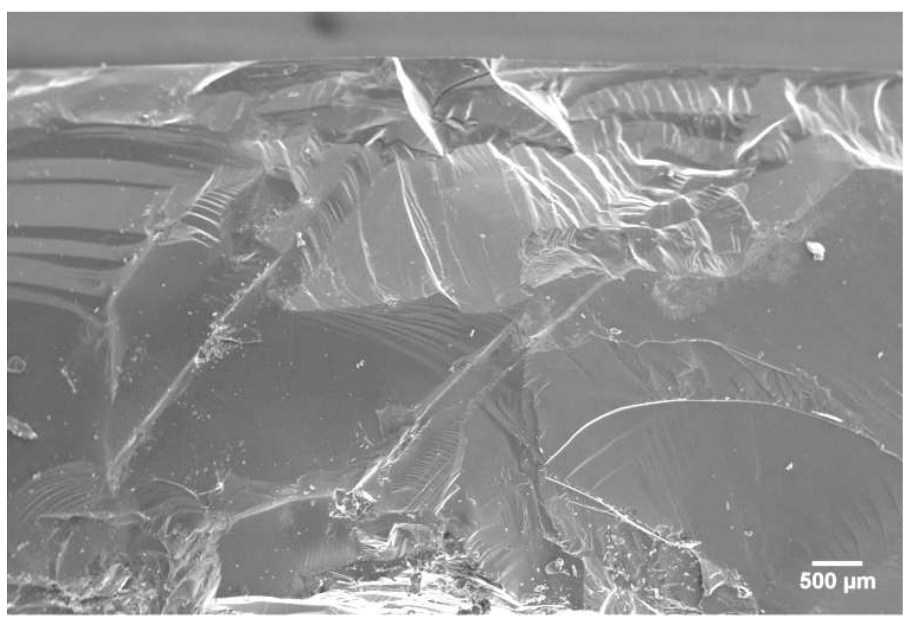

Figure 3. SEM photographs of the fracture surfaces of the middle part of the ingot obtained by the bending tests

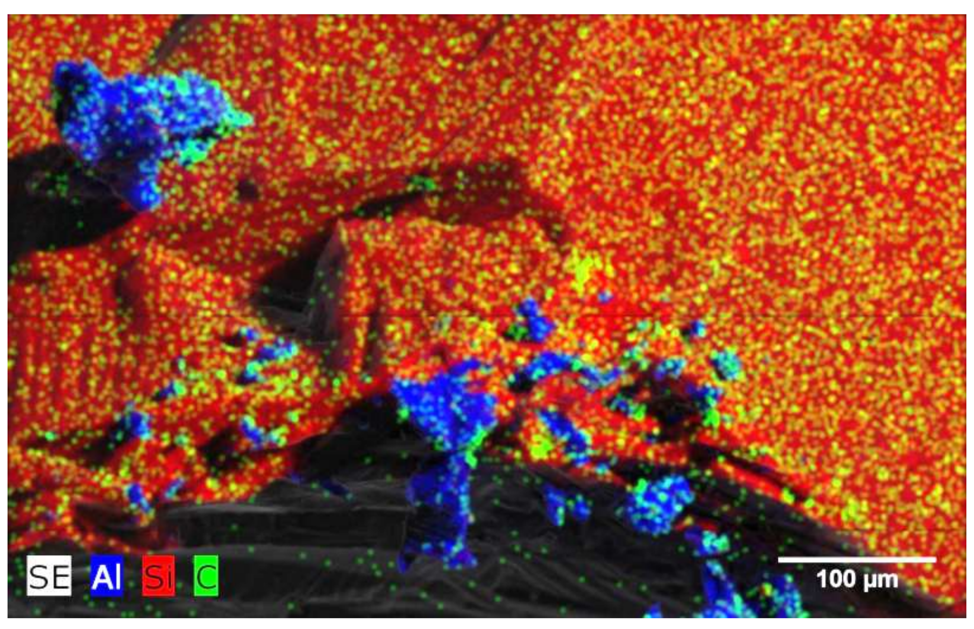

Figure 4. EDX analysis of the surface defects that originated fracture initiation

5 Conclusions

- Areas surrounding impurities are **weaker** → Fracture origin.
- Areas with high density of SiC exhibit **different *nanomechanical* properties** than the silicon matrix, may be due to the entangled residual stress fields of the SiC particles.
- Three-point and Ring-on-Ring bending tests showed **similar differences to the expected from the literature**, since the mean strength of a specimen made of brittle materials depends on its volume, its surface, its loading conditions and the flaw distribution.
- Biaxial test is **more accurate** to detect the presence of impurities.
- ***Nanohardness*, Young Modulus and Fracture toughness** don't depend on the wafer selected, just on the silicon ingot chosen, as these are **less sensible factors** to the concentration of impurities.

4th International Congress on Ceramics (ICC4)
July 15-19, 2012 | Sheraton Chicago Hotel & Towers | Chicago, Illinois, USA